UDC 678

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Selection of composite material in thin-walled structures operating at elevated temperatures

At present, of special interest are polymer composite materials reinforced with carbon fibers – carbon fiber-reinforced plastic, which have increased specific strength, rigidity, wear resistance, etc. The purpose of this research is to study the physical and mechanical properties of composite materials reinforced with carbon and aramid fibers. According to the obtained temperature dependences of σ and E, we have selected the optimal variant of the polymer composite material on the basis of a nanomodified epoxy binder reinforced with carbon fibers.

Keywords: carbon fiber-reinforced plastic, aramid filler, nanomodified binder, mechanical tests.

Introduction

Carbon plastics are a class of polymeric structural materials based on polymers reinforced with carbon fibres. These structural materials have such properties as high specific strength and stiffness, low coefficients of linear thermal expansion and friction, high wear resistance and resistance to aggressive media, thermal and radiation impact, increased thermal conductivity and electrophysical properties, high fatigue strength under static and dynamic loads [1].

The impact of fillers on the resistance and stability to high temperatures of composite materials was mentioned in the studies [2, 3]. In article [2], the degree of impact from various fillers, in particular, carbon, glass, and aramid ones, on the thermal-oxidative stability of a polymer composite material (PCM) based on an epoxy binder was studied. Carbon and aramid fillers were demonstrated to have the greatest degree of impact on the given characteristic. The authors of work [3] note that the carbon fibres noticeably affect the thermal oxidation stability and thermal stability of PCM.

Due to the above mentioned properties, the main fields of carbon plastics application are defence industries (the plastics are primarily used for aircraft and missile manufacturing).

The purpose of the present research is to study physical and mechanical properties of composite materials reinforced with carbon and aramid fibres.

Strength properties of polymer composite materials

Strength properties are one of the key parameters defining the operational purpose of the produced material. The mechanical properties of composite materials were established with regard to the thermal effect of up to 150 °C. These composite materials consist mainly of two components, a binding filler and a reinforcing one, having essentially different physical characteristics and different dependence of their mechanical properties on temperature. In most cases, the temperature resistance of the reinforcing fibres is higher than that of the binder. For this reason, the temperature dependence of such PCM physical properties as modulus of elasticity and ultimate strength, differs as defined by the loading conditions. In case of tension, the main load is borne by fibres oriented along the loading vector. Thus, the binder function is to ensure even load distribution, and then its low temperature resistance does not significantly reduce the integral characteristics of the composite material. In the process of compression, deterioration of the physical and mechanical properties of the binder at elevated temperatures causes reinforcing fibres operating in compression to lose their stability in direct proportion to the deterioration of their internal bond. Thus, in case of PCM samples, it is necessary to study the mechanical

properties of the material separately for tension and for compression.

Mechanical properties were established by means of loading the samples with a testing machine at a fixed temperature, at a constant rate of movement and at continuous sample deformation. A stress-to-deformation diagram was built based on the obtained load-to-movement dependence for the specific test temperature. The linear section of the diagram allows to define the modulus of elasticity for the sample material.

The distinctive feature of a PCM structure is the fact that its material is formed simultaneously with the structure itself, as the composite material properties are defined by reinforcing fibres netting, the ratio of components and by forming and polymerization technology. The presence of different types of compounds is crucial in ensuring structural strength. Therefore, the testing results for the simplest samples used to define physical and mechanical properties of the composite material disallow extrapolation of the characteristics to the entire structure.

Available experience in designing PCM structures with well-known characteristics can be extended to the structures of similar operational conditions made of different, new composite materials. Consequently, the behaviour of the new structure is modelled based on the existing structure of the similar type with well-known characteristics. Thus, having a detailed understanding of the operating conditions, in which a structure of close functional purpose made of a well-proven composite material is used, allows us to make a highly reliable prediction for the behaviour of a new structure made of PCM with other properties based on the comparative characteristics of the composite materials used for the structure. Therefore, defining absolute physical properties of the composite material within the simplest samples is as important as possessing their exact comparative characteristics for different PCM samples produced as per a specific process characteristic for the given option [4–6].

The possibility of modelling any lay-up with arbitrary number of layers at arbitrary reinforcement angles based on the monolayer characteristics is well known. For this purpose, samples with unidirectional lay-up are tested. At the stage of theories formation in the field of composite strength, it was the characteristics of the monolayer that were used to perform strength calculations. However, in the course of the composite industry development and accumulation of the actual data, it was established that the characteristics of the actual PCM with specific thickness and lay-up properties, moulded as per specific technology, are significantly different from the theoretical ones. This difference is due to the technology used for products manufacture. For that reason, the characteristics of a monolayer are rather an estimate than a defining feature. It is specifically for the purposes of addressing the technology impact that the tests use lay-up samples manufactured in accordance with the prescriptive technology, which is planned to be employed in the manufacturing of the actual product. In other words, lay-up samples testing provides the data required for the structure calculation of the highest precision ensuring compliance with the set margin of strength and stiffness [7–9].

Objects of research

In comparative tests, we used samples manufactured with the following binder types:

• "Alumoepoxy" is a two-component system consisting of a mixture of epoxy-diane resin and an active diluent modified with nanoparticles of aluminium oxide and a hardener, triethanolamine titanate [10–12];

• *CR-122* is a two-component epoxy system consisting of an epoxy-diane resin and an amine-based hardener;

• *CR-122* (nanomodified Ni) is a two-component epoxy system consisting of an epoxy-diane resin nanomodified with nickel and an amine-based hardener;

• *CR-122* (nanomodified Cu) is a two-component epoxy system consisting of an epoxy-diane resin nanomodified with copper and an amine-based hardener;

• *Epolam* 2092 is a two-component epoxy system consisting of a mixture of modified epoxy resins of various functions and a cycloaliphatic amine hardener.

In comparative tests, we used samples manufactured with the following types of reinforcing materials:

• *UD*-130, which is a carbon tape based on unidirectional T800 carbon fibres of high strength and stiffness as well as low density;

• CBM 56313 is a twill fabric of diamond weaving (8/3) based on aramid fibres of high strength and low density.

Samples preparation

The samples were prepared as per one of the following methods:

• compression moulding;

• moulding with an elastic diaphragm (vacuum method);

• infusion process (a type of impregnation under pressure).

Compression moulding is one of the methods used for polymer processing, in particular, for the products made of polymer composite materials. The samples of 11, 12 series were made as per this process (Fig. 1).

Application of this process requires manufacturing a special tool – a mould. The mould can be metal or composite. A blank in the form

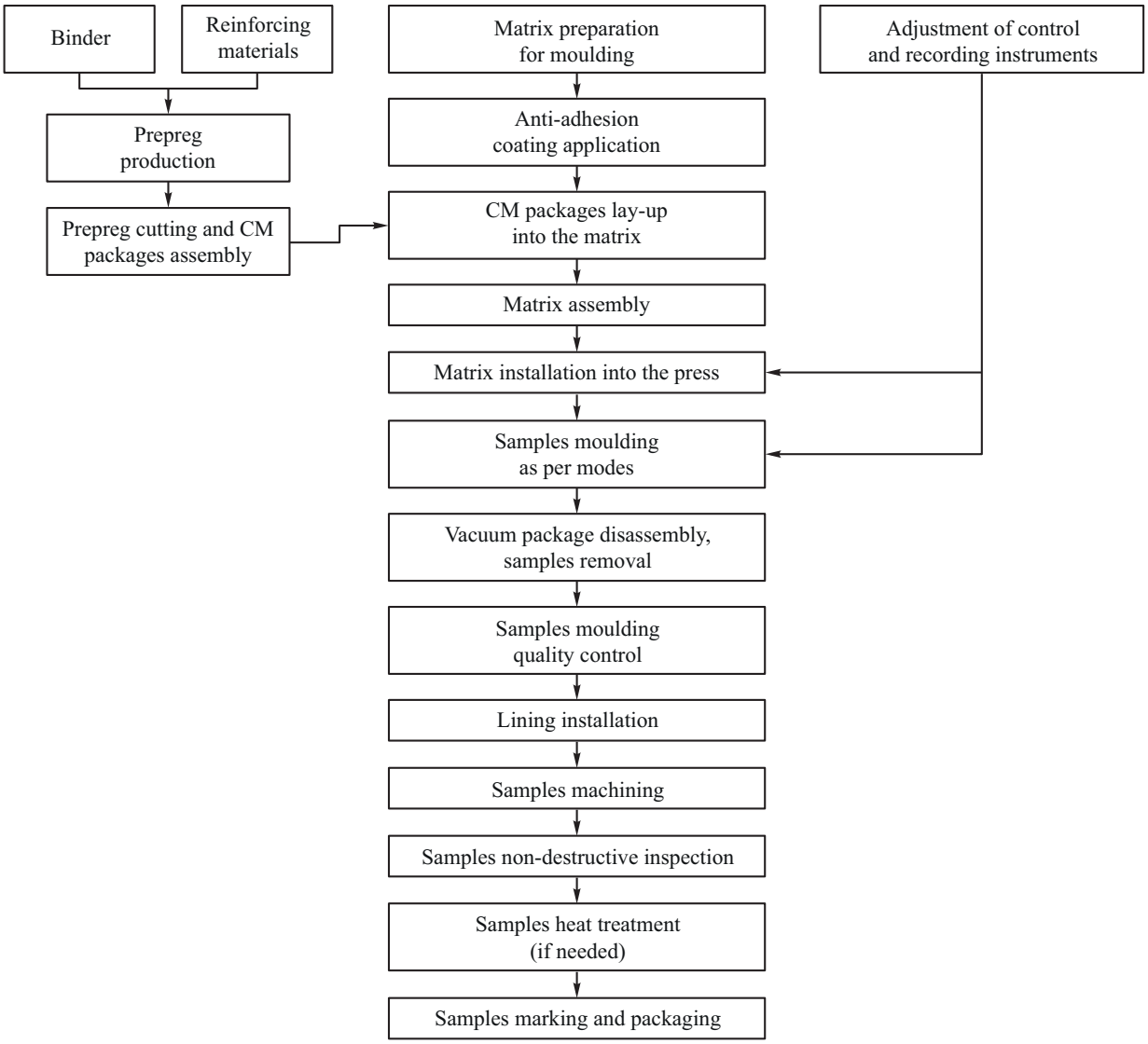

Fig. 1. Sample manufacturing process, series 11 and 12, with *Epolam 2092* binder

of a prepreg pre-impregnated with the binder is placed in the lower part of the mould, then the moulding tool is closed, put under a press with pressure directly applied onto the material, located inside the moulding cavity of the mould and then heated. At the end of the polymerization process, the product is removed from the moulding tool and machined. This method produces monolithic materials of high accuracy and reproducibility.

Nowadays more than 50 % of PCM products are manufactured by the vacuum method.

As per this process, the prepreg is laid into the inner cavity of the matrix, then the cavity is closed with a hermetically tight elastic diaphragm with air pumped out from under it. The moulding pressure (equal to the atmospheric pressure) affects the entire surface of the blank, pressing it to the internal cavity of the matrix until the main binder curing process is completed. The vacuum method is used to produce the samples of series No. 1–4, 13 and 14 with "Alumoepoxy" binder (Fig. 2).

The applied external pressure is acting on the blank material through an elastic diaphragm and performs the following functions:

- seals the layers of the reinforcing material;
- ensures deep fibre impregnation with the binder;
- pushes out air bubbles from the interlayer voids;

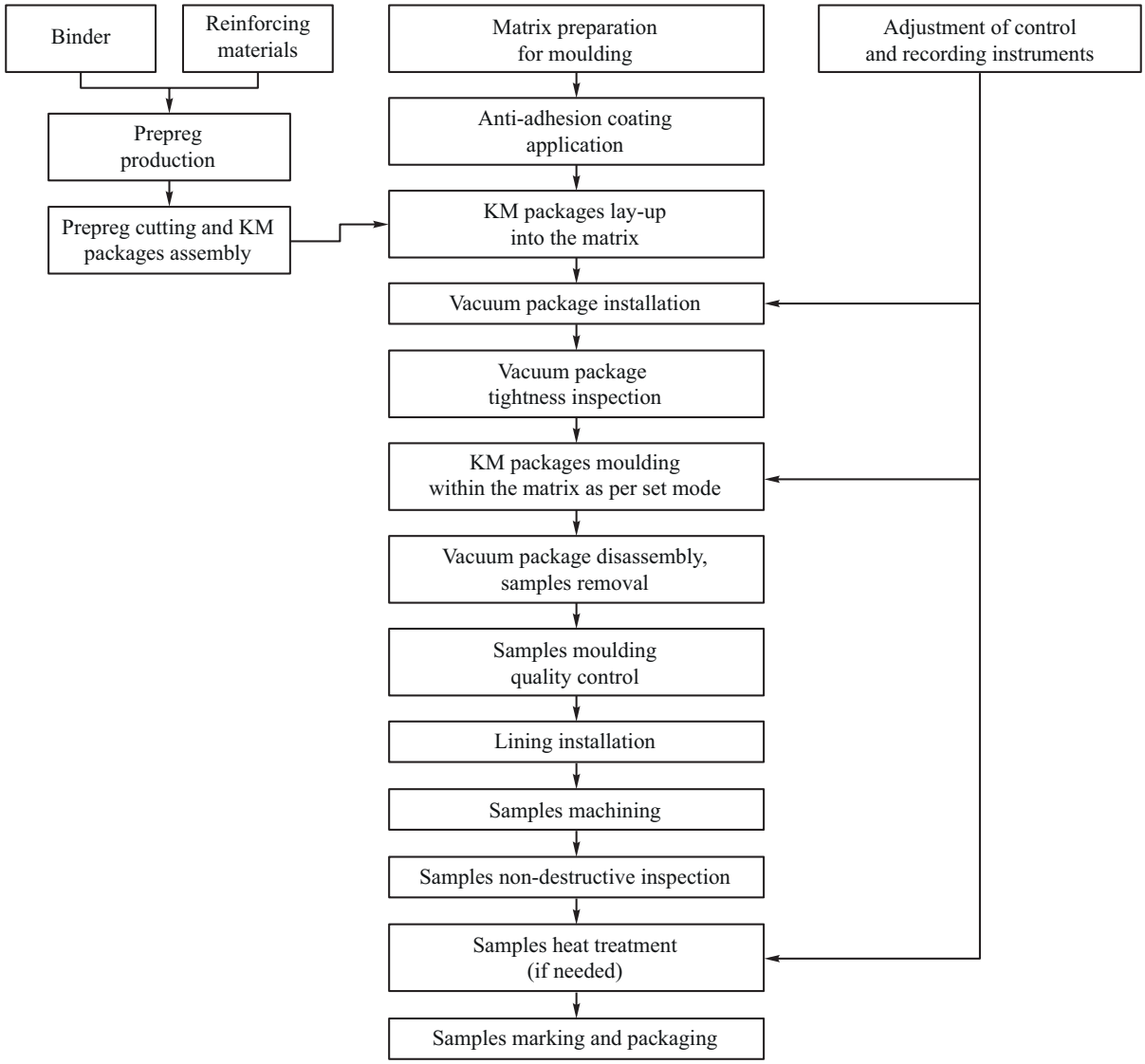

Fig. 2. Sample manufacturing process, series 1–4, 13, 14, with "*Alumoepoxy"* binder

• removes excess resin from the material layers.

Infusion process is the most modern method that allows to obtain products of complex geometric shape with minimum internal defects and voids. It is a type of pressure impregnation processes.

In this process, dry reinforcing material is laid into the internal cavity of the moulding tool, then the cavity is closed with a hermetically tight elastic diaphragm with air pumped out from under it. A binder supply system and a binder conductor in the form of a mesh are installed under the sealed diaphragm as well. Once the air is pumped from the internal cavity of the package, the binder is supplied there replacing the produced vacuum. Thus, the structure of the material containing almost zero inner air voids is formed. The moulding pressure (equal to the atmospheric pressure) affects the entire surface of the blank, pressing it to the internal cavity of the matrix until the main binder curing process is completed.

Implementing the infusion process requires the use of a special binder with low viscosity. Polymerization of the binder occurs at low temperatures of approximately 50...60 °C, then the product is removed from the moulding tool, then polymerization of residual monomer is carried out in the oven at temperatures of 100...120 °C. The samples of the series 5–10 are produced with the infusion process (Fig. 3).

The gel fraction method is used to define the optimum curing mode for each binder. It is based on the ability of the soluble (unreacted) part of the binder (sol fraction) to be washed out with solvent and is defined by the quantitative determination of the sol fraction not bound by the polymer network (of the gel fraction). Binder components were

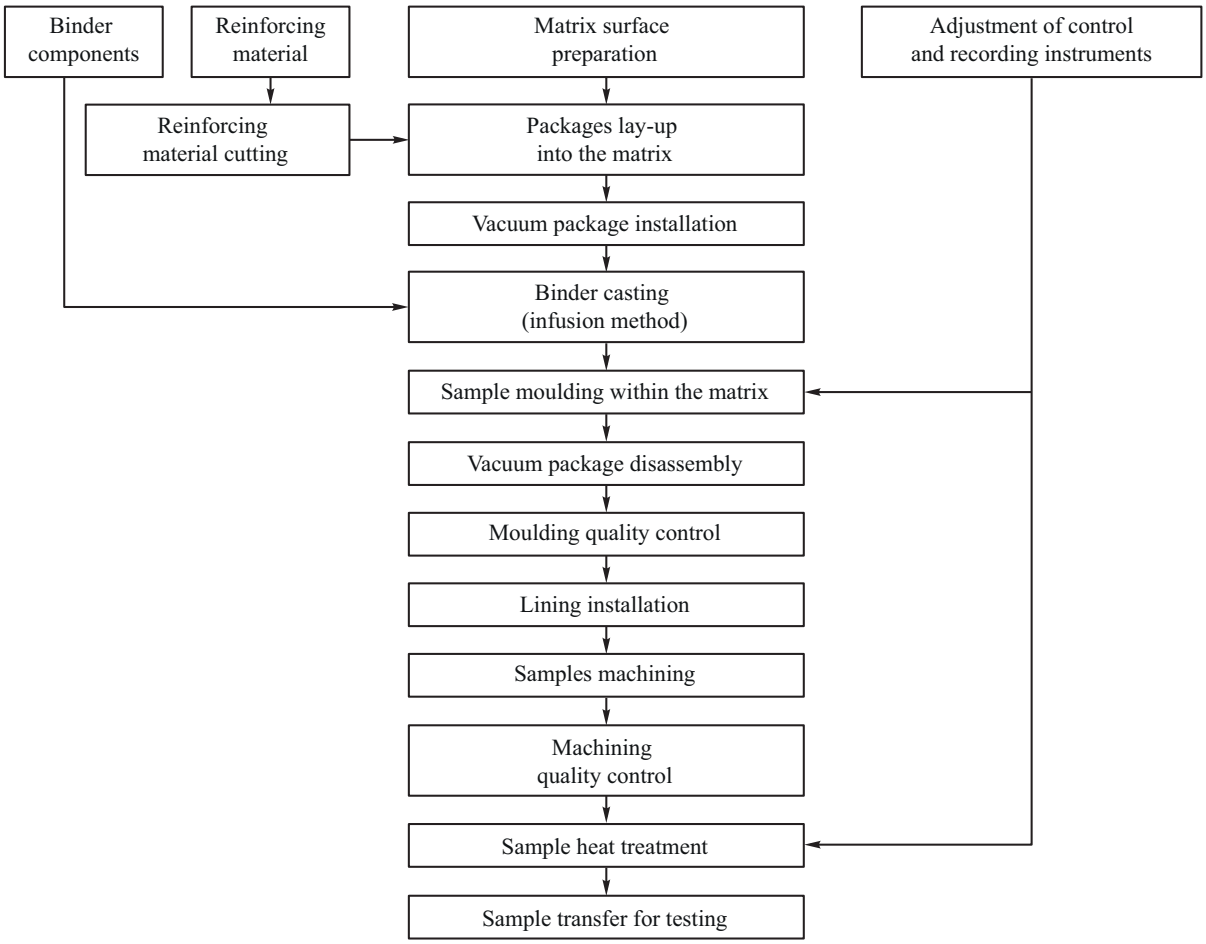

Fig. 3. Sample manufacturing process, series 5–10, with *CR-122* binder

mixed in the recommended proportions, cured and heat-treated.

The curing and heat treatment for the tests was performed as per one of the following modes (Table 1).

Table 1

Sample cure modes

Sample series	Temperature, °C	Time, h
1–4, 13, 14	100	20
	120	8
5–10	60	3
	120	12
11, 12	60	24
	120	2
	180	3

The samples obtained after curing were kept at a temperature of (20 ± 2) °C for 16 hours, then placed into the Soxhlet extractor and extracted in acetone for 6 hours. Then, the samples were dried to a constant mass and extracted again. The extraction of the soluble part of the weighed portion was considered complete if repeated results were obtained after periodical weighing.

Testing procedure

The *Zwick/Roel Z100* universal testing machine was used for samples mechanical testing.

The deformation measurement systems of the testing machine are based on the principles of contact and non-contact movement measurement at the base length of the working part of the sample. In the course of the test, the cross piece movements are recorded in time with the load values.

The loading rate (cross piece travel rate) is an adjustable parameter set prior to the testing process in accordance with the recommended values for the specific type of test. The test machine is equipped with standard *testXpert* software package, ensuring monitoring of all parameters of the machine during testing, providing a possibility to build a loading diagram in real time with recording the test results for further processing.

A temperature chamber providing the temperature range of –80...250 °C is used for testing at elevated temperature. The chamber is equipped with a heating and temperature maintenance system, controlled by two temperature sensors and a convection heater.

Processing of results and calculation of σ and *E* measured characteristics is carried out in accordance with the instructions from GOST R 56785-2015 [13] for tension tests and GOST 33519-2015 [14] for compression tests.

Summary of relative rest results by samples

The distinctive feature of testing is the requirement for preliminary heating of the sample together with grips up to the working temperature during 10–20 min for the purposes of equalizing the temperature field and preventing temperature deformations in the power circuit of the testing machine.

Compression and tensile testing of the sample types was carried out at temperatures of 20, 100, 150 °C.

Tensile tests were performed by cross piece movements measurement and with the help of a video extensometer. Measurement with a mechanical extensometer was performed at room temperature and up to 10 kN load. The given measurement was performed to evaluate the accuracy of other deformation testing methods.

The results of the tests are given in Table 2. In each series, 5 samples were tested.

It can be seen that at 150 °C all types of samples, except for two, essentially lose their strength characteristics, their $\sigma_{сж}$ does not exceed 80 MPa, which practically excludes the possibility of usage for the power elements of the structure. The mentioned exception are samples with "Alumoepoxy" binder, that showed the σ value = 135.5 MPa at 150 °C, permitting the binder to be used for high loaded structures operating at elevated temperatures. The samples with *Epolam* 2092 binder demonstrated good parameters as well, with σ kept at the level of 293 MPa, thus

Table 2

Test results

Sample No.	Sample name	Base material	Binder	Test type	Average values					
					$\sigma \cdot 10^6$, Pa			$\sigma \cdot 10^6$, Pa		
					T = 20 °C	T = 100 °C	T = 150 °C	T = 20 °C	T = 100 °C	T = 150 °C
1	Unidirectional 0°	*UD*-130	"Alumoepoxy"	Tension (E, σ)	–	–	–	147 757.5	–	–
2				Compression (E, σ)	439.4	174.5	135.5	75 444.6	63 133.0	57 044.2
3	Lay-up 0°/90°/±45°	*UD*-130	"Alumoepoxy"	Tension (E, σ)	506.0	371.4	284.6	63 824.7	45 811.0	45 381.0
4				Compression (E, σ)	354.6	115.7	78.2	58 144.2	46 873.8	38 772.0
5	Unidirectional 0°	*UD*-130	*CR*-122	Tension (E, σ)	–	–	–	126 525.0	–	–
6				Compression (E, σ)	632.8	328.0	78.7	78 714.6	70 321.3	56 606.5
7	Lay-up 0°/90°/±45°	*UD*-130	*CR*-122	Tension (E, σ)	756.9	684.3	388.9	56 823.8	53 045.8	39 316.3
8				Compression (E, σ)	402.6	271.6	44.6	44 198.0	41 798.7	34 819.5
9	Unidirectional 0°	*UD*-130	*CR*-122 Nanomod. Ni	Tension (E, σ)	714.5	268.0	80.1	72 270.2	59 745.8	49 499.4
10	Unidirectional 0°	*UD*-130	*CR*-122 Nanomod. Cu	Compression (E, σ)	640.6	216.2	70.5	69 703.6	62 596.8	50 184.0
11	Unidirectional 0°	*UD*-130	*Epolam* 2092	Tension (E, σ)	–	–	–	151 514.0	–	–
12				Compression (E, σ)	580.5	384.3	293.4	79 139.0	63 195.0	58 061.2
13	Fabric 0°		"Alumoepoxy"	Tension (E, σ)	524.0	330.5	210.2	25 509.0	17 540.6	16 403.7
14				Compression (E, σ)	127.5	49.9	49.9	37 475.2	19 024.6	20 679.4

making this binder efficient for the moulding tool manufacturing.

The effect of temperature on the modulus of elasticity is not that significant. However, when designing structures to handle stability, the temperature-related reduction of stiffness by almost 30 % shall be taken into account. In this case, the behaviour of unidirectional samples with "Alumoepoxy" binder shows no practical difference from samples with other binders. However, the characteristics of lay-up samples based on "Alumoepoxy" are considerably better than those of other binders, both at high temperatures and in cold conditions. The given fact proves better mechanical characteristics of "Alumoepoxy" binder, such as the G shear modulus, and possible manifestation of adhesive properties.

For a clearer representation of the temperature-dependent PCM characteristics, curves for $\sigma_{сж}$ and E at compression have been plotted in relative values. In Fig. 4, the percentage value of the parameter is presented along the ordinate axis with 100 % being its value at room temperature. Thus, the given graphs reflect relative deterioration of the sample properties in comparison with the initial ones depending on temperature.

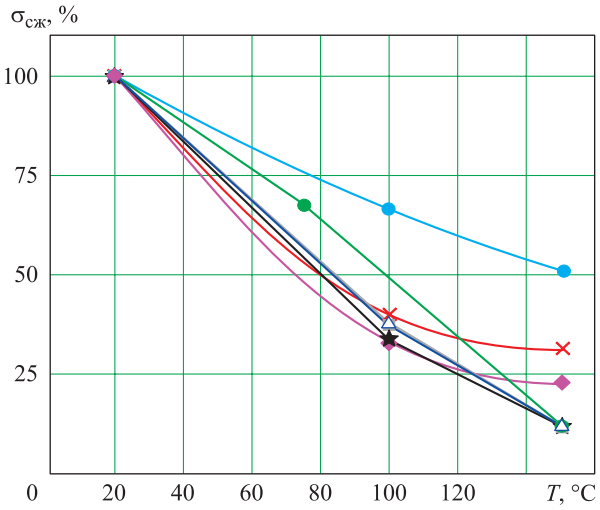

Fig. 4. Relative variation of $\sigma_{сж}$ depending on compression temperature, sample numbers: – 2; – 4; – 6; – 8; – 9; – 10; – 12

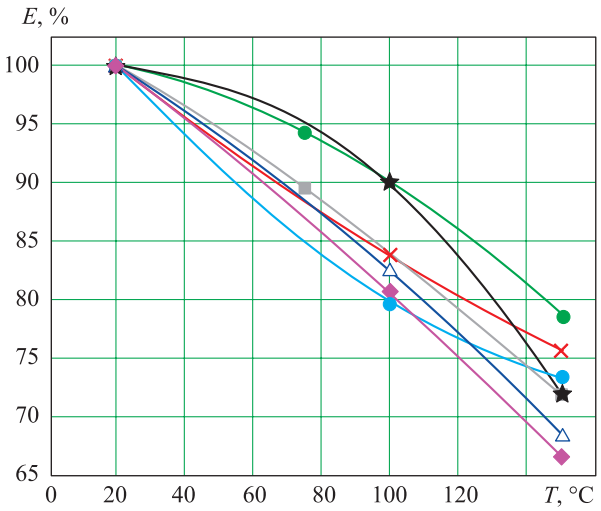

Fig. 5. Relative variation of *E* modulus depending on compression temperature, sample numbers: – 2; – 4; – 6; – 8; – 9; – 10; – 12

The temperature impact on $\sigma_{сж}$ is almost identical for all samples except for those with "Alumoepoxy" binder and Epolam 2092 binder (see Fig. 4). When heated to 150 °C, the majority of materials are clearly shown to retain their properties at 10 % of the initial level, *Epolam* 2092 at 50 %, "Alumoepoxy" at 20–30 % for lay-up and unidirectional samples, respectively.

A characteristic feature of "Alumoepoxy" binder is that it loses up to 60 % of its properties at a temperature of approx. 100 °C, but as the temperature continues to rise, the drop in properties is significantly slowing down. Such response is typical for both unidirectional and lay-up samples.

A similar diagram for the elastic modulus of *E* at compression is shown in Fig. 5.

The comparison of tensile testing results between the carbon fibre and organic plastic samples demonstrates that synthetic high-strength material (SHSM) exceeds *UD*-130 in strength by 4 % at 20 °C, but with temperature rising up to 100 °C the strength of *UD*-130 is higher than SHSM by 11 %, and at 150 °C it is higher by 26 %, i.e. *UD*-130 heat resistance in tension is significantly higher. At compression *UD*-130 characteristics are considerably higher than those of SHSM at temperatures of up to 150 °C. However, there is a difference in modulus of elasticity. At both compression and tension, *UD*-130 has 1.5-3.0 times higher elasticity than SHSM within the entire temperature range.

The diagrams for σ_p and *E* at tension in relative values are given in Fig. 6 and 7, respectively.

With consideration given to the high modulus as a defining property for thin-walled structures operating under conditions of possible loss of stability, it should be noted that using the carbon filler is more expedient than using the aramid one.

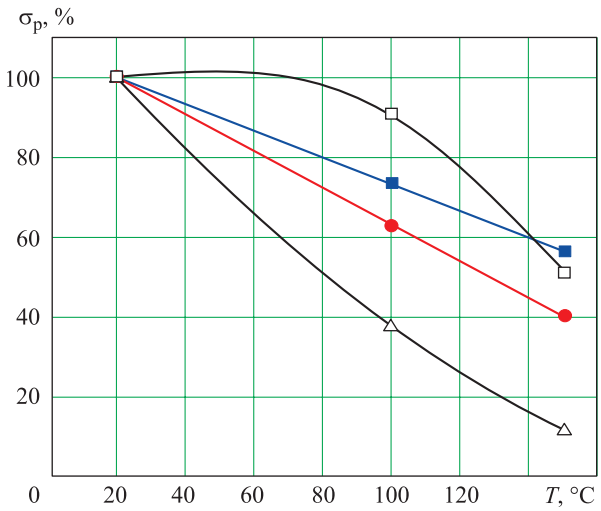

Fig. 6. Relative variation of σ_p depending on tension temperature, sample numbers: – 3; – 7; – 9; – 13

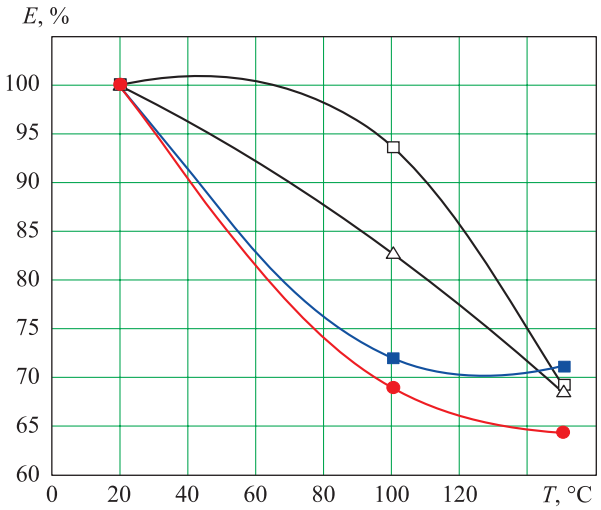

Fig. 7. Relative variation of *E* modulus depending on tension temperature, sample numbers: – 3; – 7; – 9; – 13

Conclusion

Based upon the obtained temperature dependencies of σ and *E* for carbon and aramid-based composite samples with "Alumoepoxy" binder, further work on manufacturing the designed product with account of its functional properties is feasible with the use of *UD*-130 carbon material and "Alumoepoxy" binder.

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Submitted on 29.10.2018

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Выбор композиционного материала в тонкостенных конструкциях, работающих при повышенных температурах

В настоящее время особый интерес представляют полимерные композиционные материалы, армированные углеродными волокнами, – углепластики, которые обладают повышенными удельной прочностью, жесткостью, износоустойчивостью. Цель работы – изучение физико-механических свойств композиционных материалов, армированных углеродными и арамидными волокнами. На основании полученных температурных зависимостей и выбран оптимальный вариант полимерного композиционного материала на базе наномодифицированного эпоксидного связующего, армированного углеродными волокнами.

Ключевые слова: углепластики, арамидный наполнитель, наномодифицированное связующее, механические испытания.

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